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SPACE MEASUREMENTS OF THE CONTAMINATION OF SURFACES BY OGO-6
OUTGASSING AND THEIR CLEANING BY SPUTTERING AND DESORPTION²

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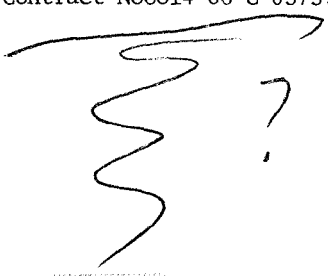
REFERENCE: McKeown, D. and Corbin, W.E. Jr., "Space Measurements of the Contamination of Surfaces by OGO-6 Outgassing and Their Cleaning by Sputtering and Desorption," ASTM/IES/AIAA Space Simulation Conference, 14-16 September 1970.

ABSTRACT: Results of the contamination of surfaces by outgassing of the OGO-6 satellite and the rates at which these surfaces are now being cleaned by sputtering and desorption after being in space for five months are given. The contamination measurements are being made with a quartz crystal microbalance to define the conditions of Al and Au surfaces used in an experiment to measure gas-surface energy transfer. It was found that the primary source of outgassing on the satellite was its two solar panels baking out in the sun. The time constant for the exponential decay of the outgassing is 1,000 hours. The maximum amount of contamination adsorbed by the surfaces exposed to the outgassing was reached after five months in orbit and is 96 mg/m² for the Al surface and 52 mg/m² for the Au surface. The contamination has a desorption activation energy of 26 kcal/g mol which falls into the energy range of materials, such as, epoxies and vacuum oils. The surfaces are now undergoing cleaning by desorption at 1.2×10^{-9} g/m²s and sputtering by upper atmospheric neutral impacts at 2.3×10^{-9} g/m²s. A method to greatly increase the cleaning rate by removal of the contamination by sputtering with upper atmospheric ions is discussed.

KEY WORDS: Surface contamination, space, outgassing, solar panels, sputtering, desorption, cleaning, ions, quartz crystal microbalance

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INTRODUCTION: With the planned orbiting of large manned space laboratories in the 70's, the space environment has taken on a new dimension. Not only must problems be considered concerned with the effects of the environment on the laboratory but also the effects of the laboratory on the environment. One area of concern is the contamination of surfaces, such as, telescope mirrors and windows by the outgassing of materials used in the construction of the craft. The contamination problem is especially troublesome because of the free molecular flow conditions that exist in the hard vacuum of space. Molecules issuing from the contamination source flow freely in straight lines to exposed surfaces. If these contaminated surfaces also point towards the sun, solar UV will compound the problem by polymerizing the oil and grease contaminants and a hard carbon glaze will form resulting in significant changes in the optical properties of the surfaces and in the accommodation coefficients for gas-surface energy transfer.

At present we are conducting a space experiment to measure gas-surface energy transfer by upper atmospheric atomic and molecular impacts to determine how satellite drag is effected by various surface materials (1)¹. The experiment is being flown on OGO-6 launched on 5 June 1969 into a polar orbit with a perigee of 397 km and an apogee of 1098 km. The experiment uses four quartz crystal probes as dual microbalances and energy transfer probes. One important part of the experiment is to monitor the rate at which material is outgassing from the satellite, how much of this material is adsorbed by the experimental surfaces, and after the outgassing rate drops to low levels, at what rate do the surfaces clean by desorption and sputtering erosion? Because the rates at which surfaces are contaminated by satellite outgassing and cleaned by desorption and sputtering are of importance to the understanding of surface contamination on other spacecraft, we would like to present here the results of measurements on the effects of a space environment on surfaces obtained from OGO-6.

INSTRUMENTATION: The flight experimental instrumentation is shown in Fig. 1 and is 11.4 x 17.8 x 19.1 cm, weighs 2.2 kg, and operates at 28 Vdc on 3.5 W. The experiment has been described in early work (1,2) and only the general operation of the instrumentation will be given here. The surfaces used in the experiment are housed in the four gold plated probes located behind the shutter wheel. The experiment is pointed along the velocity vector of the satellite into the air stream. To

1 The numbers in parentheses refer to the list of references appended to this paper.

minimize weight in the OPEP appendage of OGO-6 which has attitude control for pointing into the upper atmospheric stream, the experiment was flown in two parts. The four probe sensor package is located in the OPEP and connected by a 3 m cable to the L-shaped electronics package located in the main body of the satellite.

Four different measurements are being made on two Al and two Au surfaces. The surfaces are plated by vacuum evaporation on 15 MHz, 1.27 cm diameter, optically polished Y-cut quartz crystal sensors shown in Fig. 2. The sensors are located in gold plated probes 3.3 cm in diameter and 9 cm long, and view space through a 1.8 cm diameter window. The field of view of the surface is 0.13 steradians and the probes effectively collimate the contaminate flux reaching the surfaces.

The probes are designated A through D. Probe A is instrumented with an Al plated sensor mounted normal to the probe axis. Probe B and C have Au plated sensors mounted normal to the probe axis. Probe D has an Al plated sensor mounted at 14 degrees to the probe axis. Probes A, C, and D are pointed into the molecular stream and both contamination and energy transfer measurements are made with these probes. Probe B is pointed away from the stream by 13 degrees and the molecular stream loses its energy by multiple impacts on the wall of the probe, and only contamination measurements are made with this probe.

An eight position shutter, shown in Fig. 1, is mounted in front of the probes. Two of the 1.8 cm diameter apertures in the shutter have a 0.5 mm thick BaF₂ blank mounted over it for chopping the beam. BaF₂ is used for the shutter because its wide IR band minimized temperature changes during shutter operation. The shutter can be periodically stepped open to permit the upper atmospheric molecular stream to simultaneously impact on the Al and Au surfaces to measure gas-surface energy transfer. The shutter is programmed to remain open a total of 67% of the time and closed for 33% of the time and 67% of the outgassing flux reaches the surfaces. On every apogee pass (100 min) the frequency outputs of the probes are read out to determine the mass loading caused by contamination. The contamination measurement is made at this point in the orbit because the upper atmospheric kinetic energy flux falls below 100 mW/m², the energy transfer threshold of the probe, and energy transfer does not interfere with contamination measurements.

The probe output frequency is the beat frequency between the sensor crystal and a matched reference crystal. The beat frequency is used because it effectively cancels out ambient temperature changes and any variation in the power supply voltage driving the probe. The reference crystal is mounted in a chamber just behind the sensor crystal. The chamber front wall is 0.05 mm thick gold plated stainless steel with a 1.27 cm hole cut in it and is attached to the circumference of the sensor crystal. This does not affect the crystal oscillation because an amplitude node exists at its edge. The chamber keeps

any contamination from reaching the back surface of the sensor crystal or either surface of the reference crystal which would effect the accuracy of the measurements. The Q or quality factor of the crystals is 200,000 which assures high frequency stability from aging and any changes in the transistors used to drive the crystal oscillators. The general equation for the frequency change, ΔF , of the sensor crystal caused by mass loading (3) is

$$\Delta F = (\Delta m F_s^2 / k \rho A) + F_s(T) - F_r(T) \quad (1)$$

where Δm is the contaminant loading in g, F_s , the fundamental frequency of the crystal in Hz, k , constant of quartz = 1.66×10^6 mm, A , the motional area of the crystal in cm^2 , and ρ , the density of quartz in g/cm^3 , and $F_s(T)$ the frequency dependence of the sensor and reference crystal to ambient temperature changes. $A = 0.1 \text{ cm}^2$ and letting $\Delta F_T = F_s(T) - F_r(T)$

$$\Delta m = (2.5 \times 10^{-9} \text{ g/cm}^2 \text{ Hz}) (\Delta F - \Delta F_T) \quad (2)$$

The ambient temperature of the probes is monitored to $\pm 1^\circ\text{C}$ and $\Delta F_T = 20 \text{ Hz}/^\circ\text{C}$ for Probes A, B, and C and these probes can measure contaminant mass loading to an accuracy of $\pm 5 \times 10^{-8} \text{ g/cm}^2$ ($\pm 0.5 \text{ mg/m}^2$). The sensor in Probe D is affected to a greater degree by temperature because it is mounted at a 14° angle to the axis of the probe. Its accuracy is $\pm 2 \times 10^{-7} \text{ g/cm}^2$ ($\pm 2 \text{ mg/m}^2$).

SURFACE CONTAMINATION MEASUREMENTS: The surfaces were prepared in the laboratory by vacuum evaporation and sputtered atomically clean before instrumentation in the experiment. From the laboratory until insertion into orbit the Al surface collected 32 mg/m^2 and the Au surface 14 mg/m^2 of mass from exposure to atmospheric contamination on the ground (Los Angeles smog).

Results of contamination of Al and Au surfaces on OGO-6 are shown in Fig. 3. Data given from launch until 31 July are production data from the continuous readout of the experiment. From 1 August on, the data is "quick look" received every few days for several minutes at a time which accounts for the gaps in the measurement points.

The Al surface placed normal to the direction of the contamination flux has adsorbed approximately four times as much contamination as an Al surface mounted 14° to the flux and shows that the outgassing is external to the experiment and not internally generated. The Al surfaces have adsorbed about twice as much mass as the gold surfaces. The Al is probably gaining more mass because the outgassing material has a larger sticking coefficient for Al than Au. The Au surface in the air stream has gained a little more mass than the Au surface

shielded from the stream. One explanation for this is that the contamination layer on the surface in the molecular stream is picking up a small amount of atomic oxygen by chemisorption while the control surface shielded from direct atmospheric impacts is not reacting with O.

The contamination flux reaches a maximum when the surfaces are pointed towards either one of the two solar panels on the satellite in June and drops to near zero in August when pointed into deep space because of rotation of satellite orbit plane to the earth-sun line, and shows that the panels are the primary source of contamination.

The satellite outgassing flux consists of two main components, volatile gases such as O₂, N₂, CO₂, and H₂O and relatively low-volatile materials such as oil and paint that require a long period to outgas.

The volatile materials boil off the satellite and reach very low levels in the first few days after launch. The cloud that enveloped the satellite apparently shielded it from direct atmospheric impacts for many hours and incoming atmospheric molecules lost a significant portion of their kinetic energy by particle-particle interactions before impacting on surfaces. We were unable to detect any gas-surface energy transfer from the upper atmosphere until 72 hours after launch. Because these volatile gases are not adsorbed to any significant amount by the surfaces used in the experiment, no quantitative measurement on the flux of these particles was made.

The low volatile materials outgassing from the satellite stick to the experiment surfaces and an estimate of the flux of the low-volatile material was possible. Assuming a sticking coefficient of the flux on aluminum of 0.7 at the start of adsorption (4) and taking into account the time the shutter is closed and the view angle of the surface, the mass flow to the experiment at zero revolutions from Fig. 3 is

$$[dM/dt]_{t=0} = 9.2 \times 10^{-11} \text{ g/cm}^2\text{s} \quad (3)$$

In the International System units the flow is $9.2 \times 10^{-7} \text{ g/m}^2\text{s}$. Assuming the molecular weight averages 200 g, because low-volatile oils have large molecular weights, the outgassing flux in the vicinity of the experiment is

$$F = 2.7 \times 10^{11} \text{ part/cm}^2\text{s} \quad (4)$$

In SI units the flux is $2.7 \times 10^{15} \text{ part/m}^2\text{s}$. This flux rate decreases exponentially with time as the panels outgas. Fitting an exponential curve to the A1 curve for the first three days after launch gives a time constant of 1,000 hours for the exponential decay and

$$F = 2.7 \times 10^{11} e^{-t/10^3} \text{ part/cm}^2\text{s} \quad (5)$$

The decay in the outgassing flux is plotted in Fig. 4 from launch through January 1970.

The flux plotted in Fig. 4 is for the case of the experiment remaining fixed relative to the solar panel. Since the experiment is constantly turning to remain pointed into the air stream and the panels are turning to remain pointed at the sun, the flux reaching the face of the experiment is

$$F = [2.7 \times 10^{11} e^{-t/10^3}] [k + \cos(\theta - \pi/4)] \quad (6)$$

where F is in $\text{part/cm}^2\text{s}$, t in hours, k a constant < 0.1 to account for outgassing other than from the panels, and θ the angle between the experiment face and the solar panels. The rate of rotation of the orbit plane to the earth-sun line determines the angle, θ , between the experiment and the panels where $\theta = 4.5 \times 10^{-4} \pi t$ where t is in hours.

DESORPTION: The desorption or outgassing of materials from OGO-6, although easily measured by a quartz crystal microbalance, is very small and reflects the care with which materials were chosen in the construction of the satellite. The maximum amount of the outgassing material adsorbed by the aluminum surface was $9.6 \mu\text{g/cm}^2$ (96 mg/m^2) which represents less than 50 monolayers, assuming an average molecular weight of 200 g.

It has been pointed out that the solar panels are the primary outgassing source on OGO-6. The front surfaces are solar cells and the back surfaces of the panels are painted with ZrO_2 pigmented potassium silicate inorganic paint. Because this material has a low vapor pressure, epoxies used in assembling the panels and contamination absorbed in vacuum tests must be the contaminant sources.

This material is now deposited on the Al and Au test surfaces. The preliminary desorption rate of the material can be found when the experiment was pointed toward deep space in August. The amount of adsorbed gas, S_r , on the Au surface (4) at time t is

$$S_r = S_0 \exp(-t/\tau_r) \quad (7)$$

where S_0 is the amount on the surface at $t = 0$, and τ_r the average residence time of the gas.

$$\tau_r = \tau_0 \exp(\Delta E/RT) \quad (8)$$

where τ_0 is the vibrational period of the lattice, ΔE the desorption activation energy, R the gas constant, and T the abso-

lute temperature.

The desorption activation energy for the contamination is

$$\Delta E = -RT[\ln(2.8 \times 10^{-17}/t)(\ln S_o/S_r)] \quad (9)$$

The average temperature of the surface was 7°C and from Fig. 3 during August for Probe B: $S_o = 4.5 \times 10^{-6} \text{g/cm}^2$, $S_r = 4.1 \times 10^{-6} \text{g/cm}^2$, $t = 750 \text{ h}$, and

$$\Delta E = 26 \text{ kcal/g mol} \quad (10)$$

Griffith (4) points out that diffusion pump oils and epoxies have desorption activation energies of about 25 to 30 kcal/g mol and begin to vaporize in significant amounts at temperatures above 45°C. In full sun light the OGO-6 solar panels operate above 45°C. The contamination outgassing from the panels is then most probably epoxy assembly materials and diffusion pump oil absorbed during pre-launch vacuum tests.

RADIANT ENERGY EXCHANGE: In designing the experiment, a thermal model was made of the instrumentation to minimize the energy radiated from the surfaces generated by gas-surface interactions to maximize the temperature increase of the sensor to attain large output signals.

Radiant energy exchange between elements of a system of nonblackbodies were computed by

$$q_{ij} = \sigma F_{ij} F_{eij} A_i (T_i^4 - T_j^4) \quad (11)$$

where σ , is the Stefan-Boltzman constant, F_{ij} , the ordinary view factor for blackbodies, A_i , the area of the i'th element, T_i , the temperature of the i'th element, T_j , the temperature of the j'th element.

F_{eij} is a factor which accounts for the energy transferred between i and j where heat transfer is a function of multiple reflections between i and j themselves and other elements of the system. F_{eij} is a function of both the geometry of the system and the emittance of the various elements. The exchange of energy is directly related to the emissivity of the surfaces and would be reflected in the exchange rate measurements made during calibration.

The energy transfer sensors are calibrated periodically at the several power levels. Fig. 5 is an 878 μW calibration of the aluminum plated sensor made on 7 June 1969 soon after launch and Fig. 6 is the same level calibration made on 9 August 1969 after accumulation of 8 $\mu\text{g/cm}^2$ of contamination. A constant power was applied to the sensor for 110 sec. The probe frequency output was 1475 Hz in Fig. 5 and 1450 Hz in Fig. 6 and changed less than 2% which is the accuracy of reading out the

probe frequency. The near normal emittance of Al surface was measured in the laboratory and found to be 0.02 and has not changed significantly after being contaminated in space because the power calibration has not changed significantly.

Figs. 5 and 6 are typical of the output of the probes when energy transfer is taking place produced by molecular impacts. An exponential curve fit is made by computer of the frequency increase of the probe, $F(t)$, to find the beam energy transferred when the shutter opens.

$$F(t) = F_1(1 - e^{-t/\tau_1}) + F_0 \quad (12)$$

where τ_1 is a time constant and F_1 is the asymptotic frequency increase of the probe when the shutter opens. The constants are found by calibrating the probes in space (1).

SPUTTERING: Sputtering is the ejection of surface atoms by energetic impacts of neutral atoms or ions. For impact energies below 50 keV the interaction between the bombarding particle and target atom is treated as a hard sphere collision. For normal incidence the bombarding particle undergoes at least one collision to reverse its momentum vector before a second collision can produce sputtering. The bombarding ion strikes a target atom and rebounds in such a direction as to sputter another atom from the surface. The other possibility is that the bombarding particle displaces from its site the first atom it meets and is subsequently lost in the interstitial spaces of the lattice. Meanwhile, the displaced atom uses up its excess kinetic energy to collide with other atoms in the lattice. A certain percent of the time, this atom will strike an atom near the surface with enough energy to cause sputtering. At oblique incidence a single collision can lead directly to the ejection of a surface atom.

The sputtering yield is defined as the number of surface atoms ejected per impacting particle. The yield is dependent upon the angle of incidence, the energy, and the mass of the impacting particle and the mass of the target atom, the average binding energy of an atom in the lattice, the crystal structure of the solid, the heats of sublimation of the different crystal planes, the collision cross section of the target atoms, and the energy dissipated in the lattice as heat. Henschke (5) points out the dependence of the yield below impact energies of 100 eV on the collision radii. This dependence reveals a marked periodicity of this energy as a function of the atomic number of the target atom. More specifically, this behavior is seen to be related to the filling of the d subshells, (electronic orbital angular momentum $\ell = 2$) in the electronic structure of the elements. It is observed that for these atoms with nearly completely filled d shells, the collisions are more elastic and less energy is lost in the lattice. This results in higher

sputtering yields for these solids. Unfortunately no theoretical relationship exists to predict the sputtering yield for any arbitrary particle-surface combination and yields must be measured experimentally for particular combinations.

Rol et al (6) and Almen and Bruce (7) have proposed that the dependence of the yield on impact energy and mass is

$$\mu = \beta N \sigma(E) [4mME/(m + M)^2] \quad (13)$$

where μ is the yield in atoms/particle, β a proportionality constant to fit the experimental data, N the number density of surface atoms, $\sigma(E)$ the energy dependent scattering cross section, m the mass of the impacting particle, M the mass of the surface atom, and E the particle impact energy.

The impacting particle energy, E , is expressed in eV. From Eq. 13, the following observations can be made. The scattering cross section is known to increase with energy so yields will increase strongly with impacting energy. The yield will be maximized when the mass of the impact particle equals the mass of the surface atom ($m = M$).

Experimentally it has been found that for most particle-surface combination yields as a function of energy fall in the following ranges:

$$\begin{aligned} 0 < \mu < 0.5; 0 < E < 100 \text{ eV} \\ 0.5 < \mu < 15; 100 \text{ eV} < E < 10 \text{ keV} \\ 15 < \mu < 50; 10 \text{ keV} < E < 100 \text{ keV} \end{aligned}$$

Measurements for Au in noble gas ions in Fig. 7 illustrate the effect of impacting particles of different masses between 100 eV and 800 eV.

As the beam energies are reduced below 100 eV to 10 eV the sputtering yields fall exponentially to values of less than 10^{-3} atom/ion. It is not possible to conduct sputtering experiments at yields below 10^{-3} because laboratory beams are not intense enough to produce measureable mass losses in the few hours time required in the best vacuum systems before system impurities contaminate the surface. In the upper atmosphere, an orbiting satellite generates a molecular beam in the 10 eV range about 1,000 times more intense than is possible in the laboratory. Sputtering can then be measured on a satellite if the surface is placed in the molecular stream pointed towards space so that outgassing cannot contaminate it.

During August in Fig. 3, it was possible to determine the sputtering on OGO-6 when the surfaces were pointed into space. The desorbing mass loss rate of the Au surface shielded from impacts is 1.2×10^{-9} g/m²s. The mass loss rate for the other surface caused by sputtering and desorption at normal incidence is $M_{Al} = 3.5 \times 10^{-9}$ g/m²s, $M_{Au} = 3.5 \times 10^{-9}$ g/m²s, and M_{Al} at 14° incidence = 3.8×10^{-9} g/m²s. Subtracting the desorption mass loss rate of 1.2×10^{-9} g/m²s from the surfaces gives the

sputtering rates. The average particle flux over an orbit is 1.1×10^{17} molecules/m²s, and assuming the contamination has a molecular weight of 200 g, the sputtering yields are

$$\begin{aligned}\mu_{14^\circ} &= 3 \times 10^{-4} \text{ atom/molecule} \\ \mu_{90^\circ} &= 7 \times 10^{-5} \text{ atom/molecule}\end{aligned}$$

These yields are not for Al and Au but the contamination on the surfaces. The grazing angle of incidence produced higher yield because the impacting molecule can eject atoms without having to reverse its momentum direction.

The sputtering yields are in agreement with the Au yield, $\mu = 1 \times 10^{-6}$ atom/molecule, measured on Discoverers 26 and 32 (8) with a quartz crystal microbalance mounted on an appendage pointed continually into space. The lower yield for atomically clean Au is indicative of the higher heat of vaporization for Au than for the oil type contamination measured on OGO-6.

DISCUSSION: The measurements on OGO-6 show that outgassing of low-volatile materials which cause surface contamination is produced primarily by structures baking out in the sun such as solar panels. The contaminated surfaces can be cleaned by desorption and sputtering.

Desorption requires that the surfaces be heated to temperatures above 45°C to produce acceptable cleaning rates. The heating should be done electrically or indirectly by solar heating because if the surface is exposed to the sun, UV will polymerize the oil contamination leaving behind a permanent carbon glaze which in most cases could cause more problems for a surface than a coating of oil.

Sputtering is another cleaning method. It has a big advantage in that it can remove carbon contamination which desorption cannot. To sputter carbon, the impacting particles must transfer at least 5 eV, to the surface to break the carbon lattice bond. The neutral upper atmosphere impacts between 5 eV and 10 eV and will sputter carbon but is impractical because the yield is too low. A better source of sputtering particles is upper atmospheric O⁺ ions. Metallic surfaces would be biased to a potential of several hundred eV. A biased wire grid, 95% open, would be moved in front of a non-conductor, to accelerate the ions. Space charge would spread the ion beam and no grid shadow would form on the surface. The surface would be kept from charging up by impacting ions by directing about a 1 mA/m² flux of electrons at the surface.

During the day at altitudes below 1,000 km, the ion density is about 10^9 ions/m³ (9). An orbiting satellite sweeps out 8×10^{14} ions/m²s. At impact energies of 500 eV, the sputtering rate is about a million times greater than the rate measured at 10 eV on OGO-6. The ion sputtering cleaning rate would be about 1 mg/m²s, which would clean the 100 mg/m² of

contamination from the surfaces on OGO-6 in 100 seconds.

Above 1,000 km, ion densities will be too low for sputtering cleaning. At these altitudes an ion gun would be employed using an onboard gas supply to sputter the surfaces. The electronics already developed for use in ion engines would be a good starting point for the design of such a gun.

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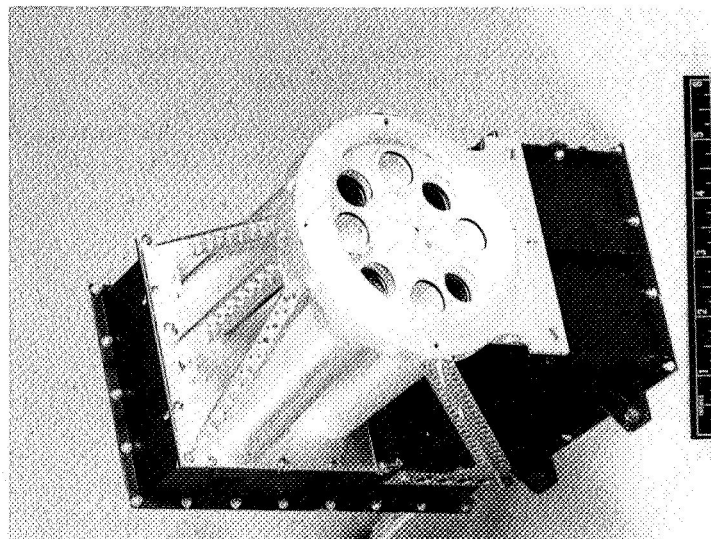


Fig. 1 : Flight instrumentation

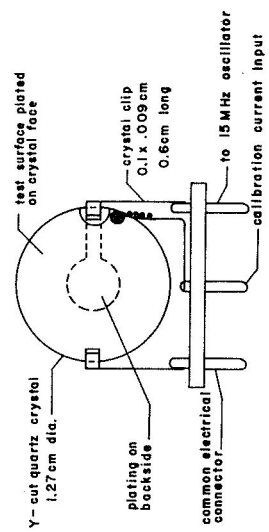


Fig. 2 : Quartz crystal sensor

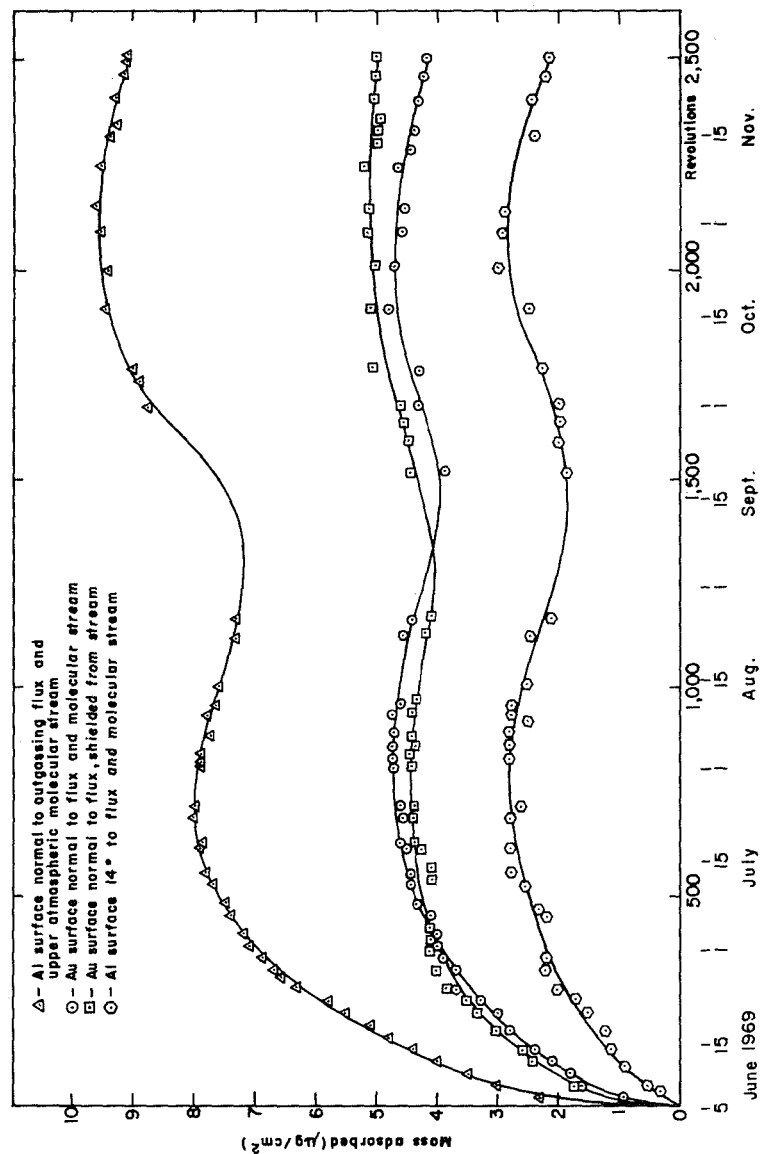


Fig. 3 Contamination loading of surfaces by outgassing of OGO-6 under various environmental conditions.

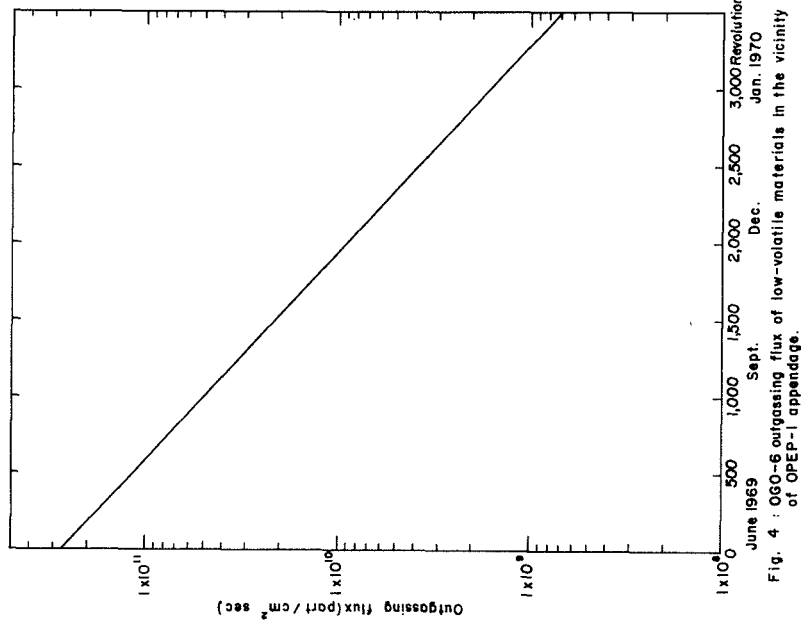


Fig. 4 : OGO-6 outgassing flux of low-volatile materials in the vicinity of OPEP-1 appendage.

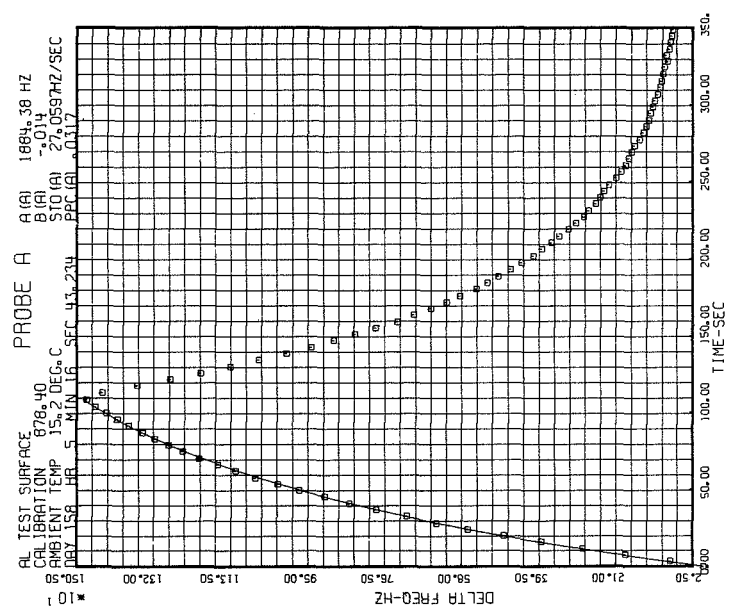


Fig. 5 : Probe A output for 878 μ W calibration with 1.8 μ g/cm² of surface contamination on 7 June 1969.

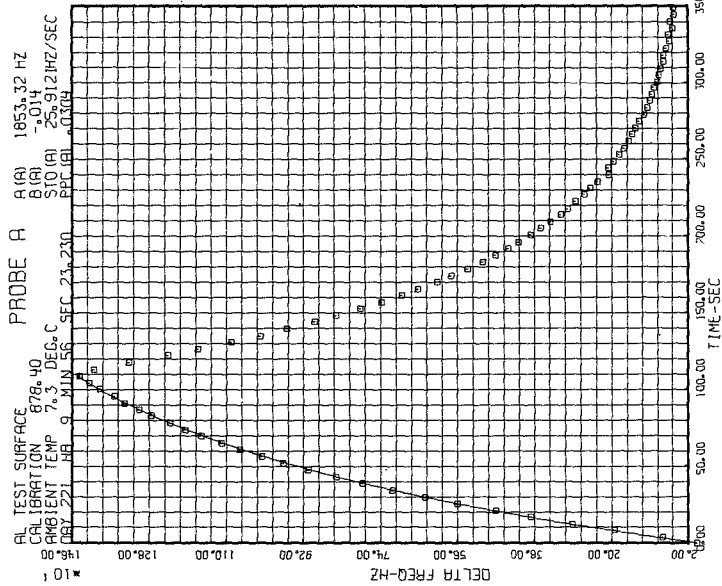


Fig. 6 : Probe A output for 878 μ W calibration with 7.7 μ g/cm² of surface contamination on 9 Aug. 1969.

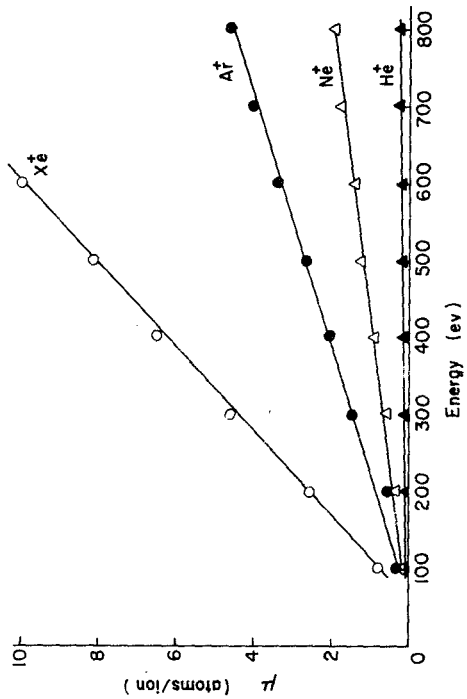


Fig. 7 : Sputtering yields of Au in noble gas ions at impact energies between 100 eV and 800 eV.